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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 : B01J 8/22 | A1

(11) International Publication Number: WO 94/16807

(43) International Publication Date: 4 August 1994 (04.08.94)

(21) International Application Number: PCT/NO94/00023

(22) International Filing Date: 26 January 1994 (26.01.94)

(30) Priority Data:
9301723.4 28 January 1993 (28.01.93) GB

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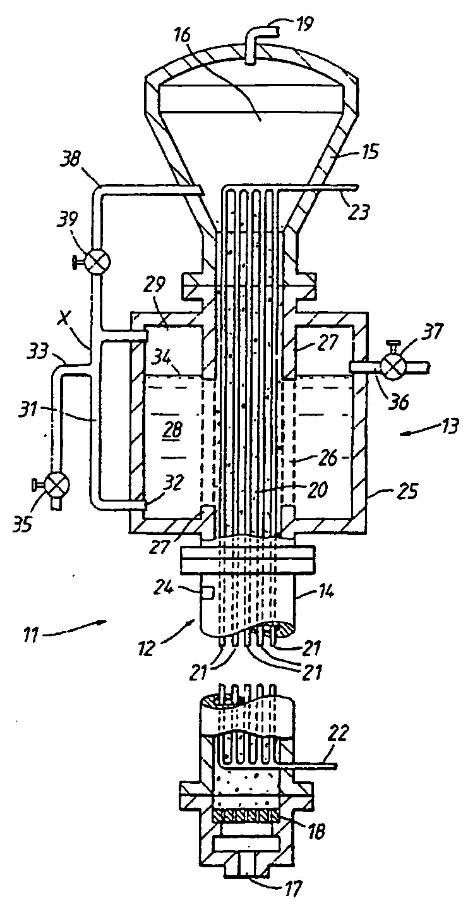
(81) Designated States: AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, LV, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA, US, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: SOLID/LIQUID SLURRY TREATMENT APPARATUS AND CATALYTIC MULTI-PHASE REACTOR

(57) Abstract

Solid/liquid slurry reaction apparatus (11) for a Fisher-Tropsch synthesis. The slurry comprises a reaction vessel (12) defining a reaction zone for the slurry phase with a volume of gas (16) above, a frit plate (18) for introducing gaseous reactants into the slurry, and a filtration section (13) arranged to separate the liquid product from the slurry. The filtration section (13) includes a housing (25), a filter element (26), an outlet (32) for the product filtrate. A tube (38) establishes fluid communication between the gas space (29) above the filtrate and gas space (16) above the slurry.



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SOLID/LIQUID SLURRY TREATMENT APPARATUS AND CATALYTIC MULTI-PHASE REACTOR

The present invention relates to a reactor for conducting a continuous multi-phase catalytic reaction and is particularly, though not exclusively, applicable to the catalytic conversion of syngas, produced by the reforming of methane, to hydrocarbon fuels, by a Fischer-Tropsch type of synthesis. Other reaction systems for which the apparatus would be suitable include various slurry reactions for the production of petrochemicals, the production of oxygenates from synthesis gas and dehydrogenation reactions. Additionally, however, the apparatus is equally useful in solid/liquid slurry treatment applications.

Three-phase catalytic reaction systems are used in a number of chemical processes and their application in the petrochemical industry appears to be increasing. Among the three-phase systems in use, there are mechanically agitated slurry reactors, and loop and bubble column slurry reactors. These employ small catalyst particles dispersed in the liquid and so in most applications, the liquid will have to be separated from the slurry to remove liquid products or for catalyst regeneration purposes. In those cases where the liquid is an inert medium, occasionally, it may have to be replaced due to degradation or the build-up of impurities.

Mechanically agitated slurry reactors are particularly convenient for batch processes due to the low mass-transfer and heat resistance. These features also make them suitable for the determination of reaction kinetics in the laboratory. A serious disadvantage and limitation of this reactor type, however, is the difficulty in the separation of catalyst particles in any continuous operation.

Commercially, it is only mechanically agitated

reactors that are used in the hydrogenation of double bonds of oils from cottonseed, soybean, corn, sunflower, etc. By employing a nickel catalyst, the products include margarine, shortening, soap and greases. The operation of bubble column slurry reactors is simple, since mechanically moving parts are avoided. Combined with the low diffusional resistance and efficient heat transfer , these reactors are attractive for many industrial processes. However, solid-liquid separation is usually performed outside the reactor in elaborate filtering and settling systems. The catalyst slurry is to be recycled to the reactor, sometimes with the use of a slurry pump. Thus, serious problems may be encountered in the continuous operation of bubble column slurry reactors.

As world oil resources diminish it is becoming more attractive to use natural gas as an energy source and methods of upgrading this to higher hydrocarbon fuels are increasing in importance.

It is therefore an object of the invention to provide a reactor which allows continuous method of conducting a multi-phase catalytic reaction which does not suffer the drawbacks of the prior art.

It is a particular object of the invention to provide such a reactor which is well suited to use in the conversion of natural gas via syngas to diesel fuel.

A recent report issued by the United States Department of Energy addressed the question of catalyst/wax separation in Fischer-Tropsch slurry reactor systems. The report concludes:

"Internal filters immersed in the reactor slurry, as used in some bench-scale or pilot-scale units, do not work successfully due to operational difficulties. A reactor with a section of its wall as a filter may be

operable for a pilot plant but is not practicable for commercial reactors. Internal filters are subject to plugging risks, which may cause premature termination of the run, and commercial plants are not allowed to take chances."

The report states elsewhere that an internal filter within the reaction slurry has been employed in a research project. However, while a flow of filtrate was initially possible by employing a pressure differential, the filter soon became clogged and it was concluded that continuous operation would not be practical and that for a commercial-scale operation, it would be necessary to perform the solid/liquid separation outside the reactor.

The present Applicants have discovered that, contrary to this teaching, it is possible to provide a continuous reaction system for a Fischer-Tropsch synthesis in which it is not necessary to perform the solid/liquid separation in an external filter unit. Furthermore, a sufficiently high flow rate of filtrate for commercial operation can be achieved.

According to one aspect of the invention, there is provided solid/liquid slurry reaction apparatus for reactions in which a liquid product is formed in a slurry phase including a finely divided catalyst in a liquid medium, the apparatus comprising: a reaction vessel defining a reaction zone arranged to accommodate the slurry phase and a volume of gas above the slurry phase; means for introducing gaseous reactants and/or other components into the slurry phase in the lower region of the vessel; a filtration section arranged to separate the liquid product from the slurry phase, the filtration section including a housing and a filter element which together define a filtrate zone, the filtrate zone having an outlet for the product filtrate; means establishing

fluid communication between the filtrate zone and that part of the reaction zone which in use is to be occupied by the volume of gas above the slurry phase; means for establishing a mean pressure differential across the filter element; in one embodiment, the means for establishing a mean pressure differential across the filter element comprises a valve and a control unit connected to the outlet of the filtrate zone. Alternatively, the filter element is arranged to be in contact with the slurry in the slurry zone and the housing at least partially surrounds the reactor vessel.

The apparatus may also be used for slightly different purposes, such as treatments of various kinds, e.g., ion exchange, purifications, removal of unwanted components (traces of impurities, discolourations).

Thus, in its broadest sense, the present invention provides a solid/liquid slurry treatment apparatus in which a liquid product is separated from a slurry phase containing finely divided catalyst in a liquid medium, the apparatus comprising: a vessel defining a treatment zone arranged to accommodate the slurry phase and a volume of gas above the slurry phase; means for introducing gaseous reactants and/or other components into the slurry phase in the lower region of the vessel; a filtration section arranged to separate the liquid product from the slurry phase, the filtration section including a housing and a filter element which together define a filtrate zone, the filtrate zone having an outlet for the product filtrate; means establishing fluid communication between the filtrate zone and that part of the treatment zone which in use is to be occupied by the volume of gas above the slurry phase; means for establishing a mean pressure differential across the filter element; and in which the filter element is

arranged to be in contact with the slurry in the slurry zone and the housing at least partially surrounds the vessel.

It has been discovered by the present Applicants that the communication between the filtrate zone and the reaction or treatment zone prevents the build-up of solid material on the filter element. [The term "reaction zone" will be used to refer to the reaction zone or the treatment zone, as appropriate.] This is believed to be achieved as follows. The turbulent motion of the slurry, as gas bubbles pass up through it, causes fluctuations or oscillations in pressure at the filter element. The fluid communication between the reaction zone and the filtrate zone facilitates or enhances these pressure fluctuations or oscillations.

Such a system is therefore relatively simple yet effective. The separation step, generally considered to be particularly problematic, is achieved without undue complication and under proper operating conditions the filter member is self-cleaning.

Preferably, the housing circumferentially surrounds the reactor vessel for at least a portion of the extent of the reactor vessel. The filter element may be provided by a portion of the wall of the reactor vessel which is composed of a filter material. In an alternative embodiment, the filter element is located outside the vessel and the vessel is discontinuous in the region of the filter element. In another alternative, the filter element is located within the vessel and the housing is constituted by a portion of the vessel wall. Preferably, the fluid communication is between the volume of gas above the slurry phase and a volume of gas above the filtrate.

The pressure differential may result from the

hydrostatic pressure arising from the slurry in the reaction vessel having a higher hydrostatic level than the filtrate in the filtrate zone. The communication between the space above the slurry in the reaction zone and the space above the filtrate in the filtrate zone prevents the build up of pressure differentials in excess of that corresponding to the hydrostatic pressure. The communication may conveniently be via a tube extending between the top of reaction zone and the top of the filtrate zone and being open to each. Preferably, the tube connecting the two volumes of gas is arranged to facilitate the escape of any gas accumulating in the upper portion of the filtrate zone.

Preferably, the amplitude or magnitude of the fluctuations or oscillations in the pressure differential across the filter element is about the same magnitude or greater than the mean value of the static pressure differential. Preferably the mean pressure differential across the filter element should be kept at a rather low level, typically less than 6 mBar (600Pa). If the mean pressure differential is below a critical vale (6 mbar in case of the exemplified system), the filter is self cleaning. With slightly higher values, build-up of a cake of particles on the filter surface will occur, as one would expect for a filter, and the capacity will gradually decrease. This cake will disappear if the flow through the system is reversed (backflushed), and the original capacity will be regained. With even higher values, the catalyst particles may penetrate into the filter. If this happens, the decreased capacity may be permanent, and increased capacity by backflushing the filter may not be possible. The fluid communication tube may, in addition to effecting the communication between the gas phase above the slurry and the internal parts of

the filtration section, also provide an easy escape route for gas which may have penetrated the filter element and which otherwise would have become entrapped in the filtrate zone.

Gaseous products or components may be allowed to escape by any convenient means such as a separate outlet from the reaction vessel or simply via the fluid communication tube. Experiments carried out suggest that if the tube is closed or severely choked, the filter element would rapidly become clogged. Of course, the fluid communication tube will set a limit to the pressure drop across the filter element and thus as stated, prevent unwanted and damaging pressure build-ups, which might well otherwise occur when there is a considerable pressure drop between internal parts of the reaction vessel and the outlet side.

Preferably, the filter element comprises a fine meshed screen, helically wound threads, fine vertical threads or sintered metal particles. Preferably, the surface of the filter element which is in contact with the slurry is rendered smooth.

The filter element material and catalyst are preferably selected so that the maximum hole or pore size in the filter element is of the same order of magnitude as the catalyst particle size. The particle size is preferably not less than half the pore size.

The means for introducing gaseous reactants or components may comprise any suitable means such as a bubble cap plate, a plurality of nozzles, a frit plate, etc, preferably located at the bottom of the reaction vessel. The reactants may be CO and H_2, for example from the reforming of natural gases, and the products may be methanol and higher hydrocarbons.

The pressure fluctuation value may be of the order

of the pressure differential, for example from 10 to 200% of the pressure differential. The actual value of the pressure differential may be from 1 to 100 mBar, preferably 2 to 50 mBar.

The vessel is preferably provided with an inlet and/or an outlet for liquid reactants or components and preferably also an outlet for gas in its upper part. The apparatus may also include means for flushing the filtration section and the filter elements.

Preferably, the outlet from the filtrate zone is arranged to provide a constant level for the filtrate in the filtrate zone. In one embodiment, the outlet from the filtrate zone comprises an inverted U-shaped pipe whose top section defines the liquid level in the filtrate zone. In an alternative embodiment, the outlet from the filtrate zone includes a weir within the filtration section which defines the liquid level in the filtrate zone. In a further embodiment, the outlet from the filtrate zone comprises an upwardly extending pipe within the filtrate zone which is open at a level which defines the liquid level in the filtrate zone.

Preferably, the reaction vessel is provided with means for heat transfer. This may comprise a plurality of vertically positioned tubes intended for circulation of a heat transfer medium.

In a preferred embodiment, the fluid communication between the gas spaces above the slurry and filtrate zones is by means of a communication tube. In an alternative embodiment, the wall of the reaction vessel terminates and the filtration section housing forms a dome over the top of the reaction vessel whereby the volume of gas above the slurry and the volume of gas above the filtrate are in direct communication.

In order to accommodate an increased production

rate, a higher effective filter area may be provided. This may be achieved by adopting a more intricate or complex filter element profile.

Alternatively, this may be achieved by providing more than one filtration section. In one embodiment , the filtration sections include vertically spaced filter elements and housings which may or may not partly overlap. In an alternative embodiment however there may be plurality of vertically spaced filtration sections. Preferably, the respective filtration section housings are in the form of flanged pipe section which are screwed e.g. bolted together to form a rigid structure.

The invention is particularly well adapted for use in a method of converting natural gas (methane) to higher hydrocarbon fuels which involves initially converting the natural gas into synthesis gas, either by steam reforming, partial oxidation, a combination of the two, or otherwise reforming the methane to produce carbon monoxide and hydrogen, subjecting the CO and H_2 to catalytic conversion by a Fischer-Tropsch synthesis to form higher hydrocarbon fuels such as liquid paraffin waxes, and subsequently separating and/or cracking these products to produce the required range of hydrocarbons.

When diesel fuel is produced in this way it is vastly superior to conventional diesel in terms of its quality and properties. Firstly, it contains no sulphur, which is important from an environmental point of view. Secondly, it has a very high cetane number and can therefore be blended with lower grades of diesel fractions in order to produce a product which meets premium range standards. Thirdly, it contains virtually no harmful compounds that generate soot when burned and needs fewer additives for problem free use at low temperatures.

The invention may be carried into practice in various ways and some embodiments will now be described by way of example with reference to the accompanying drawings, in which:

Figure 1 is a schematic section through a three-phase slurry reaction apparatus for performing a method in accordance with the invention;

Figures 2 and 3 are simplified schematic sections through a part of the filtration section showing two alternative systems for achieving a constant filtrate level;

Figure 4 is a view similar to Figures 2 and 3 showing an alternative location for the filter elements

Figure 5 is a partial perspective sketch of part of the filtration section showing another possible location for the filter element;

Figure 6 is a schematic horizontal cross-section showing an alternative filter arrangement;

Figure 7 is a simplified view similar to Figure 1 showing an alternative embodiment;

Figure 8 is an enlarged vertical cross-section of the filtration system of a further embodiment;

Figure 9 is a partial vertical cross-section of a still further embodiment; and

Figure 10 is a graph showing the results of an experimental example.

As shown in Figure 1, the reaction apparatus 11 includes a reactor vessel 12 and a filtration section 13. The reactor vessel 12 includes a generally tubular section 14 and above this, an inverted cone-shaped portion 15. The tubular section 14 defines the slurry zone 20 in which a slurry of finely divided catalyst in a liquid medium of eg product hydrocarbon is accommodated. The cone-shaped portion 15 acts as an

expansion chamber to prevent the slurry from foaming over and defines a gas space 16 above the reaction zone. The cone-shaped portion 15 may contain additional means (not shown) for breaking up or reducing foam formation.

At the bottom of the vessel 12, there is a gas inlet 17 and a gas distributor 18 through which gas can be introduced into the slurry zone. At the top of the vessel 12 there is a gas outlet 19 from the gas space 16. A series of heat transfer tubes 21 are located within the reactor vessel extending between a common inlet 22 and a common outlet 23 for a heat exchange medium. The apparatus 11 will be controlled by a large number of transducers, controllers valves, pumps etc, one of which (a pressure or temperature sensor) is indicated by way of example at 24.

The filtration section 13 comprises an annular housing 25 which surrounds the vessel 12 just below the cone-shaped portion 15. Within the housing 25, a part of the vessel wall is composed of sintered metal and thus constitutes a filter element 26. Non-porous parts 27 of the vessel wall extend into the housing 25 at the top and bottom of the housing. The housing 25 and vessel wall effectively define a filtrate zone 28 and above it, a gas space 29.

An outlet from the filtrate zone 28 serves as a constant level device for the filtrate. A pipe 31 extends upwards from an outlet opening 32 near the bottom of the housing 25. A horizontal connection section 33 defines the level 34 of the filtrate in the filtrate zone 28 and extends downwards to an outlet valve 35. The valve 35 is opened to empty the accumulated liquid product in the downward leg of the pipe. Of course, the downward leg may be replaced by a holding tank for the liquid product. The outlet pipe 31 is filled with liquid

product between the opening 32 and the horizontal section 33.

A communication tube or pipe 38 connects the two gas spaces 16 and 29. The tube 38 has a valve 39. The communication tube 30 is also connected to the pipe 31 thus providing fluid communication between the gas spaces 16, 29 and the outlet pipe 31. The housing 25 also has an inlet 36 near the top with a valve 37.

In operation, gaseous reactants are introduced into the slurry of catalyst and liquid product via the gas distributor 18, maintaining the catalyst particles in suspension. The correct temperature for reaction is maintained by the various sensors eg 24 and the heat transfer system 21,22,23. Liquid product filters through the filter element 26 into the filtrate zone 28. This is encouraged by a pressure differential across the filter element caused by a hydrostatic head as a result of the difference in level between the slurry and the filtrate. The level 34 of the filtrate is maintained constant by the vertical position of the horizontal section 33 of the outlet pipe 31.

The turbulent motion of the slurry helps to prevent the build-up of any filter cake and tends to avoid the filter element 26 becoming clogged with catalyst particles by causing fluctuations or oscillations in the pressure across the filter element 26 where the valve 39 is left open.

Gaseous products and any unreacted reactant gases are removed via the outlet 19. Any build-up of gas above the filtrate in the space 29 is avoided by the presence of the communication tube 38.

The filtration section 13 can be flushed, either by a suitable gas such as synthesis gas or a suitable liquid such as purified product, by opening the valve 37 and

closing the valves 35 and 39. This forces the flushing fluid back through the filter element 26.

During normal operation, a portion of the catalyst would be removed and replaced either by new or regenerated catalyst. For reasons of clarity, the apparatus for this purpose has not been shown in Figure 1 though it is to be understood that any standard system for doing so could be employed.

An alternative constant level system for the filtrate is shown in Figure 2. Here, an annular weir 41 is provided within the housing 25. The filtrate product collects between the filtrate element 26 and the weir 41 and overflows the weir. Thus, the top of the weir determines the filtrate level 34. An outlet 42 for the filtrate product is located in the housing 25 beyond the weir 41. With this arrangement, the filtrate zone is reduced in volume and so the linear velocity of the filtrate is increased for a given volumetric flow rate. This may have an enhanced cleansing effect on the filtration section as a whole.

A further embodiment of constant level device for the filtrate is shown in Figure 3. In this case the level 34 is set by a vertical outlet pipe 51 which extends upwards in the filtrate zone.

In the embodiment shown in Figure 4, the filter element 103 is located within the reactor wall 14. Thus, the filtration section housing is constituted by a portion 104 of the reactor wall 14. The filtrate and any gas which may have passed through the filter element 103 will leave the filtration section 105 through a pipe 106 connected to the communication tube 38 and to an outlet 107 via a valve 108 controlled by a control unit 109. Any sediment which may collect at the bottom of the filtrate section may be flushed out through a pipe 110 by

opening a valve 111 when required. Settling and flushing of sediments may be intentionally achieved (in this and other embodiments) by the appropriate dimensioning of the unit and by suitably designing the outlet.

In the embodiment shown in Figure 5, the filter element 136 is located outside the reactor wall 14 but is otherwise housed with an external housing 25 as in Figure 1. The reactor wall 14 is interrupted in the region of the filter element 136 by bars 131 which allow the slurry to contact the filter element 136.

If the production rate is greater than the filter element 26 can accommodate, the capacity can be increased by increasing the effective filter area. One way of achieving this would be to replace the annular filter element 36 with a filter made up of a series of planar elements 61 as shown in Figure 6.

Alternatively two (or more) filtration sections may be employed one above the other. Figure 7 shows such an arrangement in which there are three vertically spaced filtration sections 71,89,91. Each has a respective filter element 72, 82 and 92; an annular housing 73, 83 and 93; a filtrate zone 74, 84 and 94; a filtrate product outlet 75,85,95. Each outlet 75,85,85 includes an inverted U-shaped portion and in each case, the top of the S-shape defines a constant level for the filtrate in each respective zone 74,84,94. Communication between the gas spaces above the filtrate zones 74,84,94 and the gas space 16 above the slurry zone 20 is provided by communication pipes 76,86,96 from each of the filtrate zones 74,84,94 which are connected to a common manifold 131. The manifold 131 is connected to the gas space 16 via a valve 132. The manifold 131 is also connected to those product outlets 75,85,95. Back flushing is carried out by shutting the vale 132 and the valves in lines 75,

85, 95, 76, 86, 96 and opening nitrogen inlet valves 77, 87 and 97.

In order to maintain the pressure differential at a value below 6 mBar in an experimental diethyl benzene/17% alumina slurry system, it has been found that the vertical extent of the respective filter element 72,82,92, should not exceed approximately 70-75cm. However, this would probably vary with different slurry systems according to the hydrodynamic properties of each particular slurry system. With separate filtrate level regulation for each filtration section, it is possible to obtain the desired pressure differential under these conditions. It will be appreciated that the need for a different filtrate level with a different filter element position arises from the difference in density between the filtrate and slurry.

A similar alternative arrangement is shown in Figure 8. In this embodiment, there are three vertically spaced filter elements 72,82,92. However, in place of three separate housings, there is a single outer housing 98 and two inner annular walls 88,78, extending upwards and outwards from between the three filter elements 92,82,72. The walls 88,78 terminate at different levels, thus determining the their respective constant filtrate levels 89,79. The constant level 99 of the filtrate associated with the filter element 92 is determined by the position of the outlet 101. Thus, in use, the filtrate cascades over the walls 78 and 88 into the space defined by the housing 98 and the leaves via the common outlet 101.

In this embodiment, there is a common gas space 29 above the filtrates. Communication with the gas space 16 is direct in the sense that the reaction vessel wall 14 terminates and the gas space 16 is defined by a dome 102 formed as a continuation of the outer housing 98. The

dome 102 has an outlet 19 (not shown). It will be appreciated that such an arrangement could be used with the embodiment of Figure 1 if only one filter element were employed.

Figure 9 shows another embodiment in which a series of filtration sections 113 are used. Since the filtration sections 113 are identical in size and shape only one will be described. The filtration section comprises an outer cylindrical wall 114 joined to the reaction vessel wall through flanges 115. Again, a filter element 116 is provided by a porous portion of the vessel wall. Thus, a filtrate zone 117 is defined. Each filtration section 113 has a flushing fluid inlet 122 with a valve 123, connected to a common flushing fluid supply pipe 124.

A liquid outlet from the filtration zone 117 is provided by a tube 118 having a valve 119 controlled by a control unit 120. A communication tube 121 is fitted to the upper part of the filtration zone 117 and the gas space 16 above the slurry (see Figure 1) via a common communication tube 125. The open end of the common communication tube 125 extends into the space 16 above the slurry and is fitted with a valve 126, which is generally kept open during normal operation. Alternatively, each of the communication tubes 121 may be fitted with valves (not shown).

The communication tube 121 will allow any gas which would otherwise be trapped in the upper part of the filtration zone 117, to escape the gas space 16 above the slurry via the common communication tube 121 and the valve 126.

The control unit 120 connected to the valve 119 controls the flowrate of the liquid product from the filtration zone 117 in such a manner as to establish the

mean pressure differential across the filter element 116. In effect, the control unit will maintain the hydro-static pressure head on the filtrate side of the filter element 116 relative to the hydro-static pressure in the slurry phase, by maintaining the height of the liquid column in the communication tube 121 within a fixed range. For this purpose, the control unit 120 may be provided with means (not shown) for detecting the liquid or pressure level in the communication tube 121. Thus, the valve 119 will drain liquid product from the filtration zone 117 when an upper limit is detected by the sensory means of the control unit 120. This drainage will continue until lower limit is reached, at which point the control unit 120 will close the valve 119 and the level of the liquid will start rising in the communication tube 121 until the upper limit is reached and the cycle is repeated. A continuous flow of product from the filtration zone 117 is of course also conceivable.

The hydrostatic pressure at a particular level 127 in the slurry zone is equal to the product of the depth of the slurry H_s and the density of the slurry ρ_s. Similarly, the hydrostatic pressure of the filtrate in the filtrate zone 117 at the same level 127 is given by the product of the height H_f of the discharge level 128 above the level 127 and the density of ρ_f of the filtrate. The level 128 is therefore set by the height of H_f to satisfy the equation:

$$H_f\rho_f = H_s\rho_s - \Delta P$$

where ΔP is the small pressure differential required across the filter element 116. ΔP would be given by $\Delta H_s\rho_s$ where ΔH_s is the additional slurry hydrostatic head responsible for the small pressure differential.

While this relationship has been addressed in relation to the embodiment of Figure 9, it will be appreciated that it is, of course, generally applicable in all cases.

The vertical oscillatory motion of the filtrate in the pipe 121 enhances the tendency to dislodge catalyst particles adhering to the filter element 116 and helps to prevent agglomeration and the formation of a filter cake. By suitable dimensioning of the various pipe diameters etc, it may even be possible to increase this effect by taking advantage of any resonance phenomena that may arise within the system due to the turbulent movement of the different phases.

The filtration section 113 may be flushed by means of the flushing fluid inlet pipe 122 and valve 123. When the outlet valve 119 and common communication tube valve is 126 (or alternatively, an individually fitted communication tube valve - not shown) is closed, the flushing fluid passes back through the filter 116. However, if it is desired to flush the filtrate zone 117 to remove any sediment which may have settled, the outlet valve 119 is opened, which allows a more rapid flow of the flushing fluid.

It will be appreciated that with regard to the apparatus shown in Figure 9, with careful design of the components making up the filtration sections 113, the entire reaction apparatus may be assembled from a small number of similar parts. Furthermore, these components may be standard items which are generally available, such as standard tubing, flanges, valves etc. In particular, by dimensioning the flanges of the filtration sections to be the same as the flanges associated with the filter elements 116 and by employing identical cylindrical wall sections 114, assembly and disassembly would become very

simple while the structure as a whole would be strong and rigid. The main function of the cylindrical wall sections would be to contain the fluids within the reactor and to provide rigidity in the overall structure.

Above a certain total pressure, the required dimensions of the external parts (such as, the outer cylindrical walls 114, and in particular the flanges and their securing means) of the reactor shown in Figure 9 may become inconveniently large and impractical. Under such circumstances the entire reactor vessel assembly may be encapsulated within an external pressure hull (not shown).

The invention will now be illustrated further in the following experimental example.

Experiments in a laboratory unit with a column similar to that shown in Figure 1 have been carried out. The length and diameter of the unit were 1250mm and 55mm respectively. The filter member used had a mean pore size of 30 um, and a length of 200mm.

A heat transfer oil supplied by Monsanto, consisting of diethylbenzene (95%) and minor amounts of isopropyl benzene and secbutyl benzene, and alumina (20 wt%) were used as the slurry phase. The alumina particles had initially a cut-off at 53um. The experimental conditions in the example were shown as follows:

pressure	1 bar
temperature	20 - 25°C
superficial gas velocity	6 cm/s

After an initial start-up period, the separation capacity became approximately constant at a level of 750 - 800 kg/m^{2}h. The results are given in Figure 10.

Figure 10 shows the measured separation capacity (kg/m^{2}h) as a function of time on stream from the example

described above. The calculated pressure differential i.e. the driving force for the separation is also shown in Figure 10. The mean value of this Figure is approximately 5 mbar.

After 40 days on stream only a slight decrease in separation capacity was observed, and it was concluded that a separation capacity of at least 700 kg/m^2h can be achieved for extended periods of time.

CLAIMS

1. Solid/liquid slurry treatment apparatus (11) in which a liquid product is separated from a slurry phase (20) containing finely divided catalyst in a liquid medium, the apparatus comprising: a vessel (12) defining a treatment zone arranged to accommodate the slurry phase (20) and a volume of gas above the slurry phase (20); means (17,18) for introducing gaseous reactants and/or other components into the slurry phase (20) in the lower region of the vessel (12); and a filtration section (13) arranged to separate the liquid product from the slurry phase (20), the filtration section (13) including a housing (25) and a filter element (26) which together define a filtrate zone (28), the filtrate zone (28) having an outlet (32) for the product filtrate; characterised by means (38) establishing fluid communication between the filtrate zone (28) and that part (16) of the treatment zone which in use is to be occupied by the volume of gas above the slurry phase (20); and means (31,32,33) for establishing a mean pressure differential across the filter element (26); and in which the filter element (26) is arranged to be in contact with the slurry in the treatment zone and the housing (25) at least partially surrounds the vessel (12).

2. Apparatus as claimed in Claim 1, characterised in that the housing (25) circumferentially surrounds the vessel (12) for at least a portion of the extent of the vessel (12).

3. Apparatus as claimed in Claim 1 or Claim 2, characterised in that the filter element (26) is provided

by a portion of the wall of the vessel (12) which is composed of a filter material.

4. Apparatus as claimed in Claim 1 or Claim 2, characterised in that the filter element (136) is located outside the vessel (12) and the vessel (12) is discontinuous in the region of the filter element (136).

5. Apparatus as claimed in Claim 1, characterised in that the filter element (103) is located within the vessel and the housing is constituted by a portion (104) of the vessel wall.

6. Apparatus as claimed in any preceding Claim, characterised in that, in use, the said fluid communication (38) is between the volume of gas (16) above the slurry phase and a volume of gas (29) above the filtrate.

7. Apparatus as claimed in Claim 6, characterised in that the said fluid communication is by means of a tube (38) connecting the two volumes of gas.

8. Apparatus as claimed in Claim 7, characterised in that the tube (38) connecting the two volumes of gas is arranged to facilitate the escape of any gas accumulating in the upper portion of the filtrate zone.

9. Apparatus as claimed in Claim 6, characterised in that the wall of the vessel terminates and the filtration section housing (98) forms a dome (102) over the top of the vessel whereby the volume of gas above the slurry and the volume of gas above the filtrate are in direct communication.

10. Apparatus as claimed in any preceding Claim, characterised in that the means for establishing a mean pressure differential across the filter element (103) comprises a valve (108) and a control unit (109) connected to the outlet of the filtrate zone.

11. Apparatus as claimed in any preceding Claim, characterised in that the outlet (31,32,33; 41,42; 51) from the filtrate zone is arranged to provide a constant level for the filtrate in the filtrate zone.

12. Apparatus as claimed in Claim 11, characterised in that the mean pressure differential across the filter element (26) is achieved by means of a difference in level between the slurry in the slurry zone (20) and the filtrate (34).

13. Apparatus as claimed in Claim 11 or Claim 12, characterised in that the outlet from the filtrate zone (28) comprises a pipe section having a portion (31,33) which defines an inverted U-shape, the top part (33) of the inverted U-shape defining the liquid level (34) in the filtrate zone (28).

14. Apparatus as claimed in Claim 11 or Claim 12, characterised in that the outlet from the filtrate zone (28) includes a weir (41) within the filtration section which defines the liquid level (34) in the filtrate zone (28).

15. Apparatus as claimed in Claim 11 or Claim 12, characterised in that the outlet from the filtrate zone (28) comprises an upwardly extending pipe (51) within the filtrate zone which is open at a level which defines the

liquid level (34) in the filtrate zone (28).

16. Apparatus as claimed in any preceding Claim, characterised in that the filter element (26) comprises a fine meshed screen, helically-wound metal threads or sintered particles of metal or a ceramic material.

17. Apparatus as claimed in any preceding Claim, characterised in that the surface of the filter element which is in contact with the slurry is rendered smooth.

18. Apparatus as claimed in any preceding Claim, characterised by heat transfer means (21,22,23) within the vessel (12).

19. Apparatus as claimed in Claim 18, characterised in that the heat transfer means comprises a plurality of vertically arranged tubes (21) within the vessel (12) for the circulation of a heat transfer medium.

20. Apparatus as claimed in any preceding Claim, characterised by a gas outlet (19) from the upper part of the treatment zone.

21. Apparatus as claimed in any preceding Claim, characterised by means (37) for flushing the filtration section and the filter element (26).

22. Apparatus as claimed in any preceding Claim, characterised by its plurality of filtration sections (71,81,91).

23. Apparatus as claimed in Claim 22, characterised in that the filtration sections (71,81,91) include a

vertically spaced filter elements (72,82,92) and housings (73,83,93) which at least partly overlap.

24. Apparatus as claimed in Claim 22 or Claim 23, characterised in that the respective filtration section housings are in the form of flanged pipe sections (114,115) which are secured together to form a rigid structure.

25. Apparatus as claimed in any preceding Claim, characterised in that the reaction apparatus is provided with an external pressure hull.

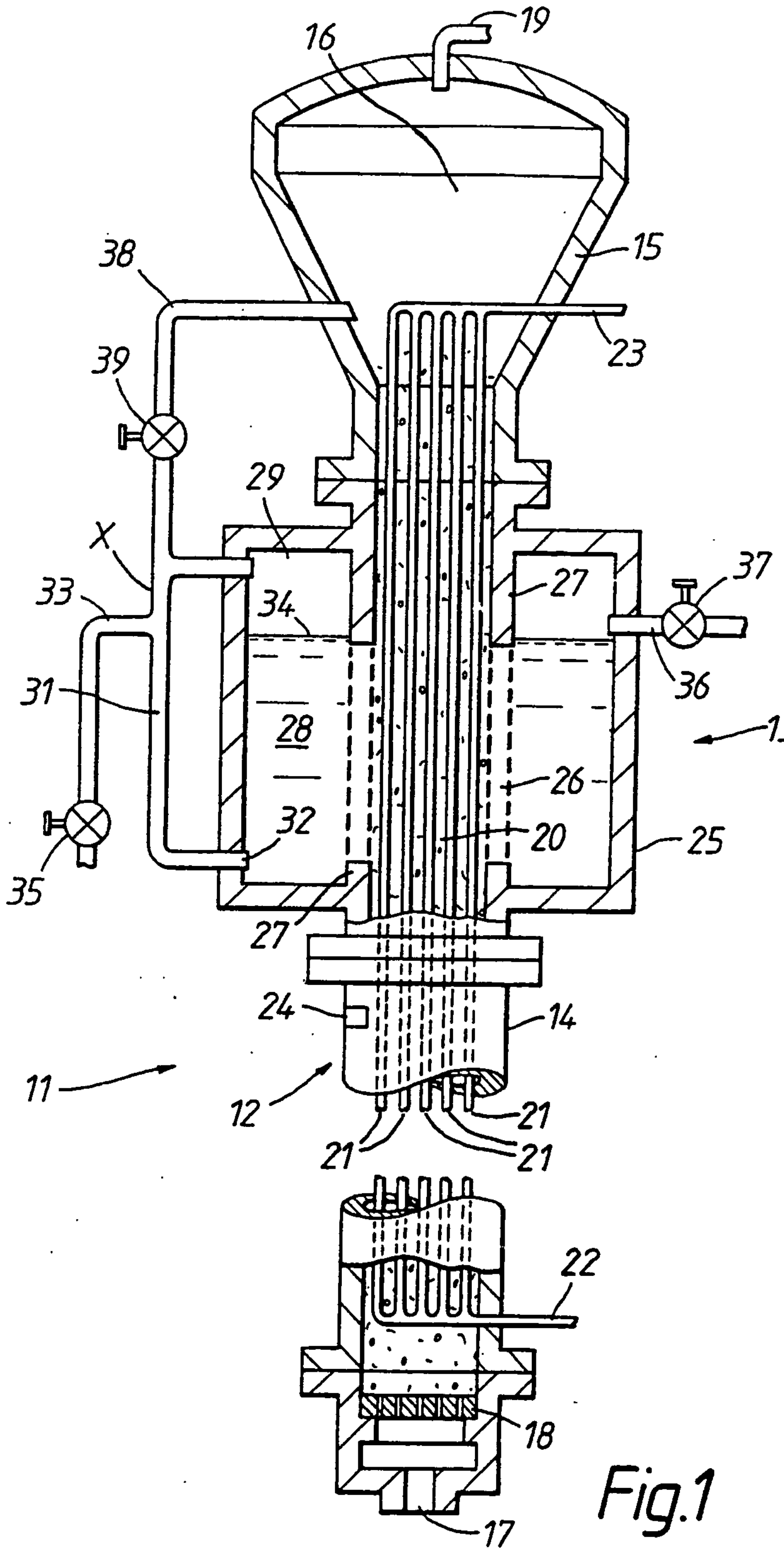
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Fig.1

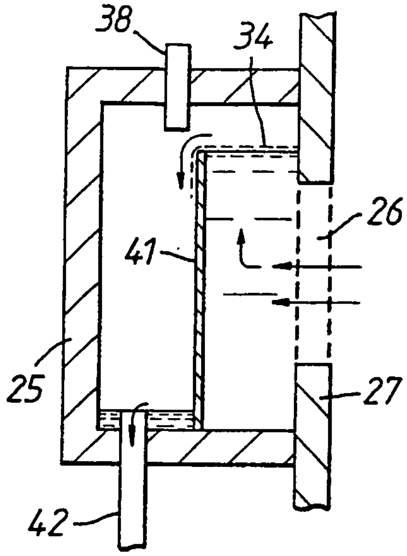

Fig.2

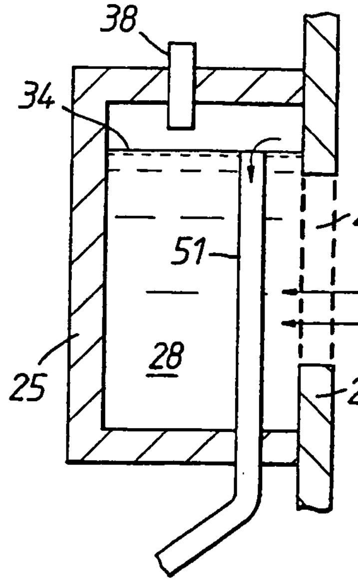

Fig.3

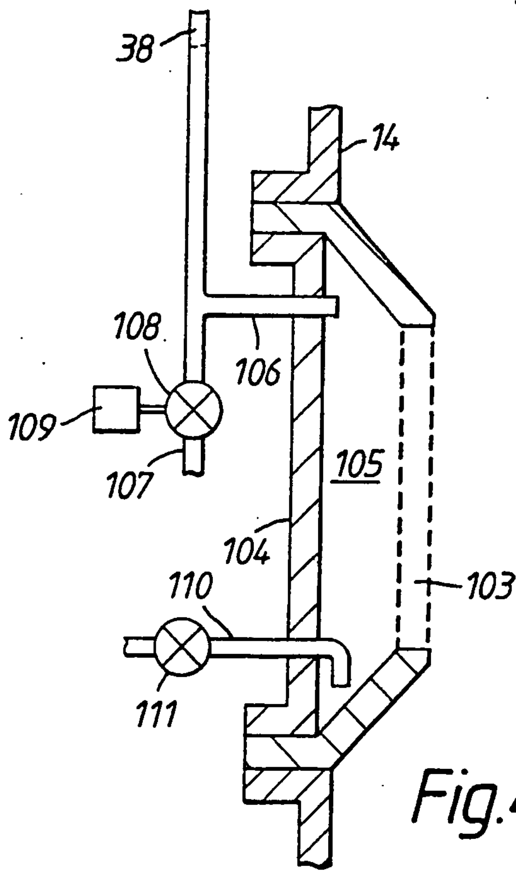

Fig.4

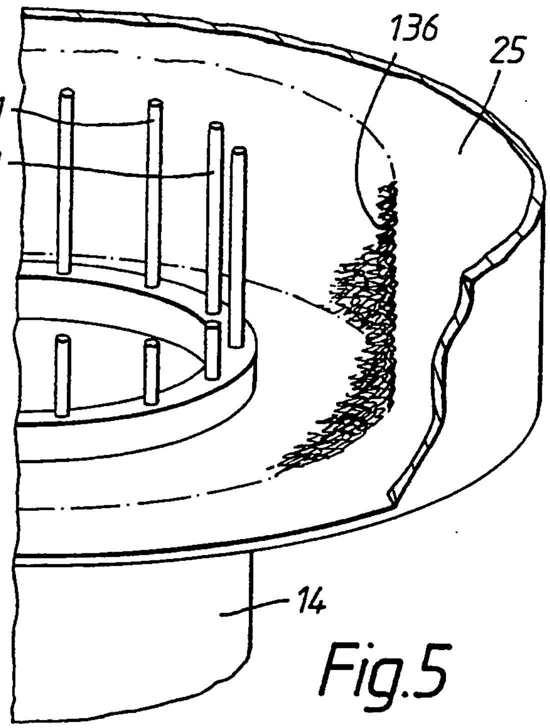

Fig.5

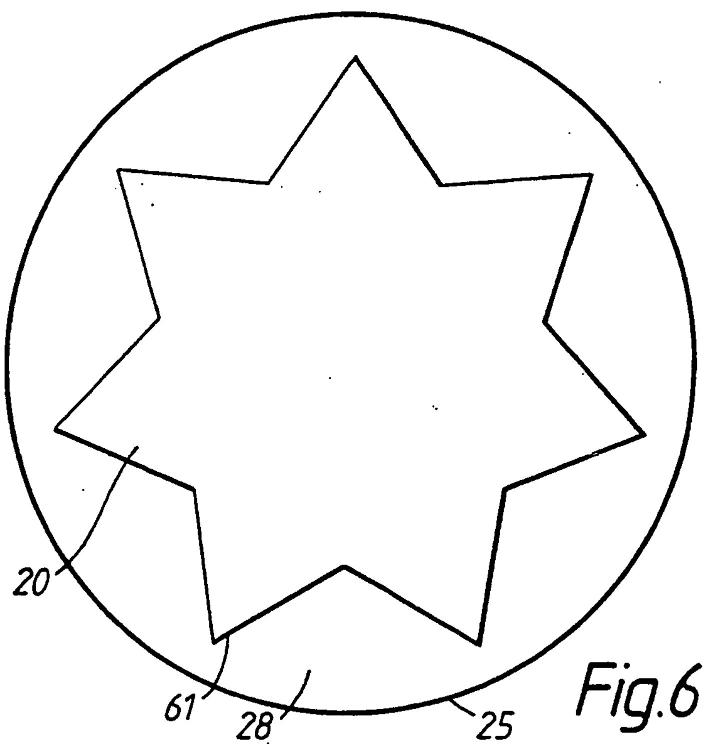

Fig.6

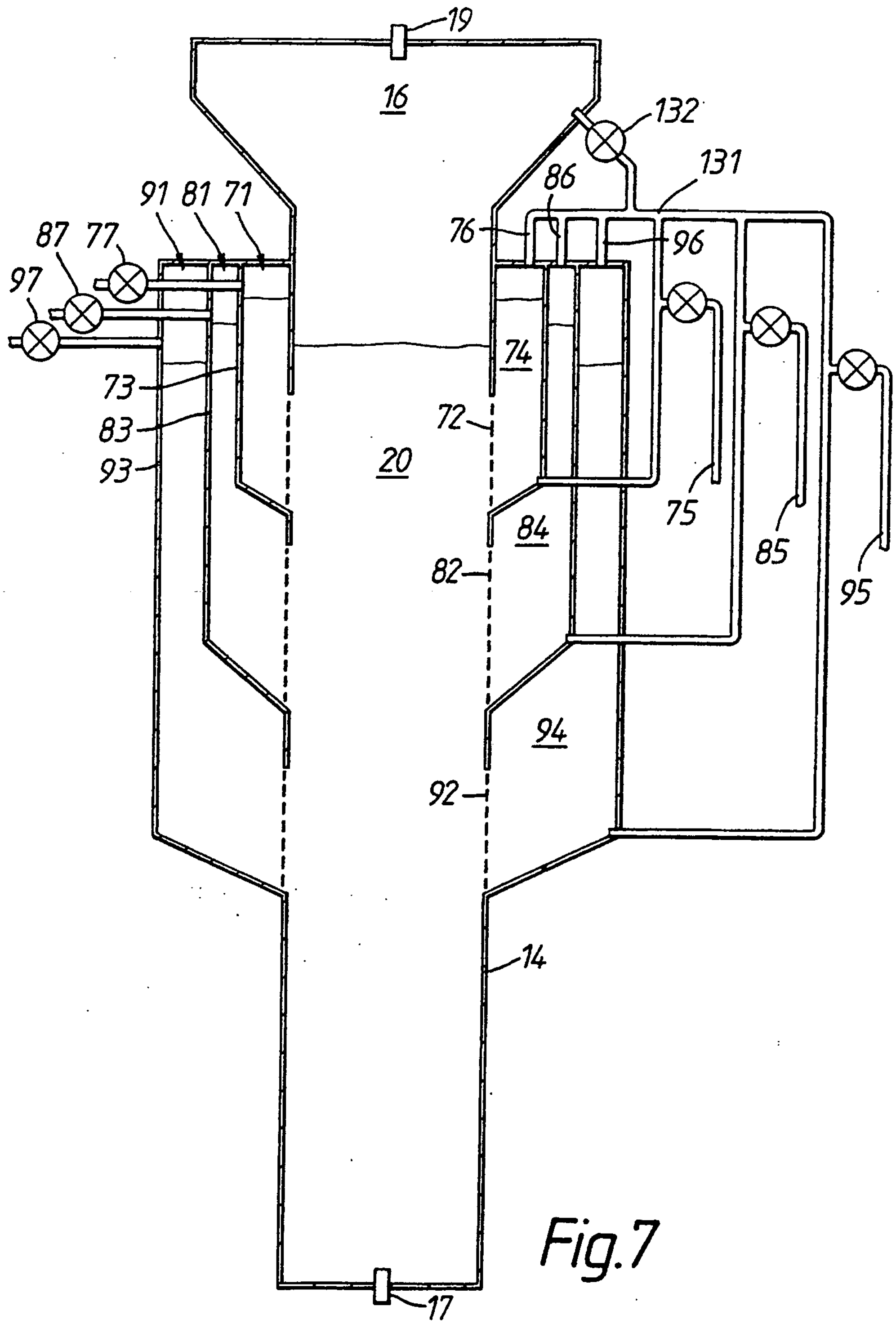
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Fig.7

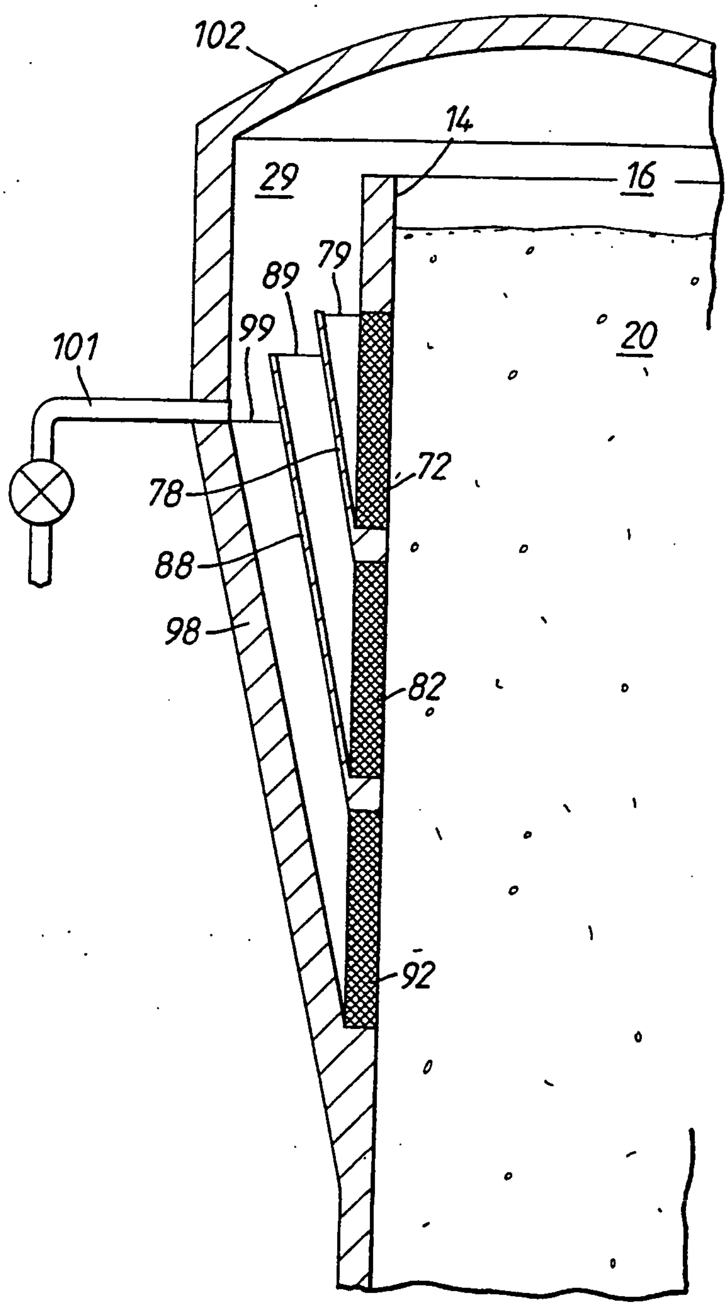

Fig.8

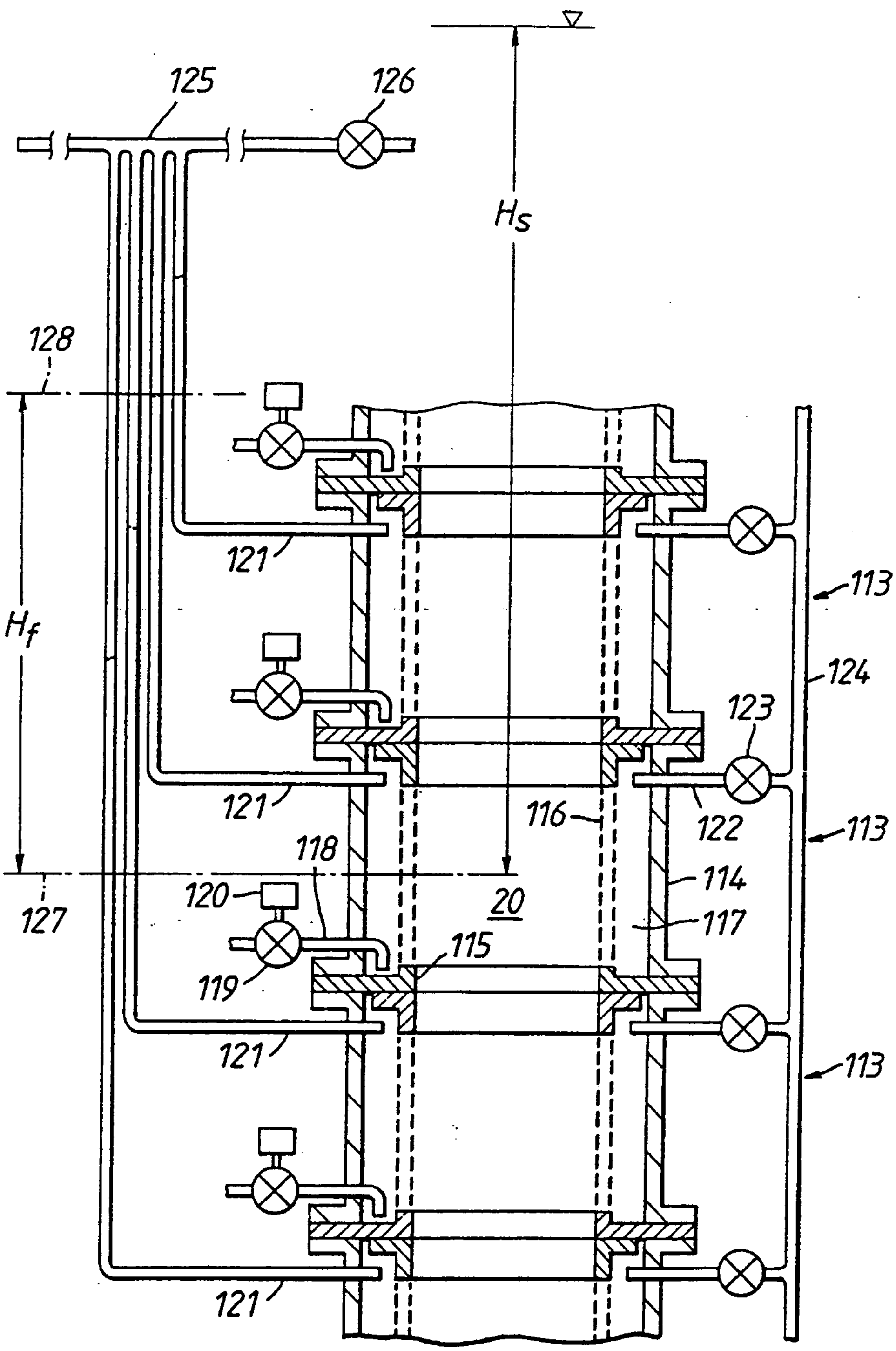
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Fig.9

SEPARATION CAPACITY, INVERTED SLURRY VALVE
20 cm LONG SINTER SECTION, GAS VELOCITY: 6cm/s
MONSANTO HEAT TRANSFER OIL, 20 % ALUMINA BY WEIGHT

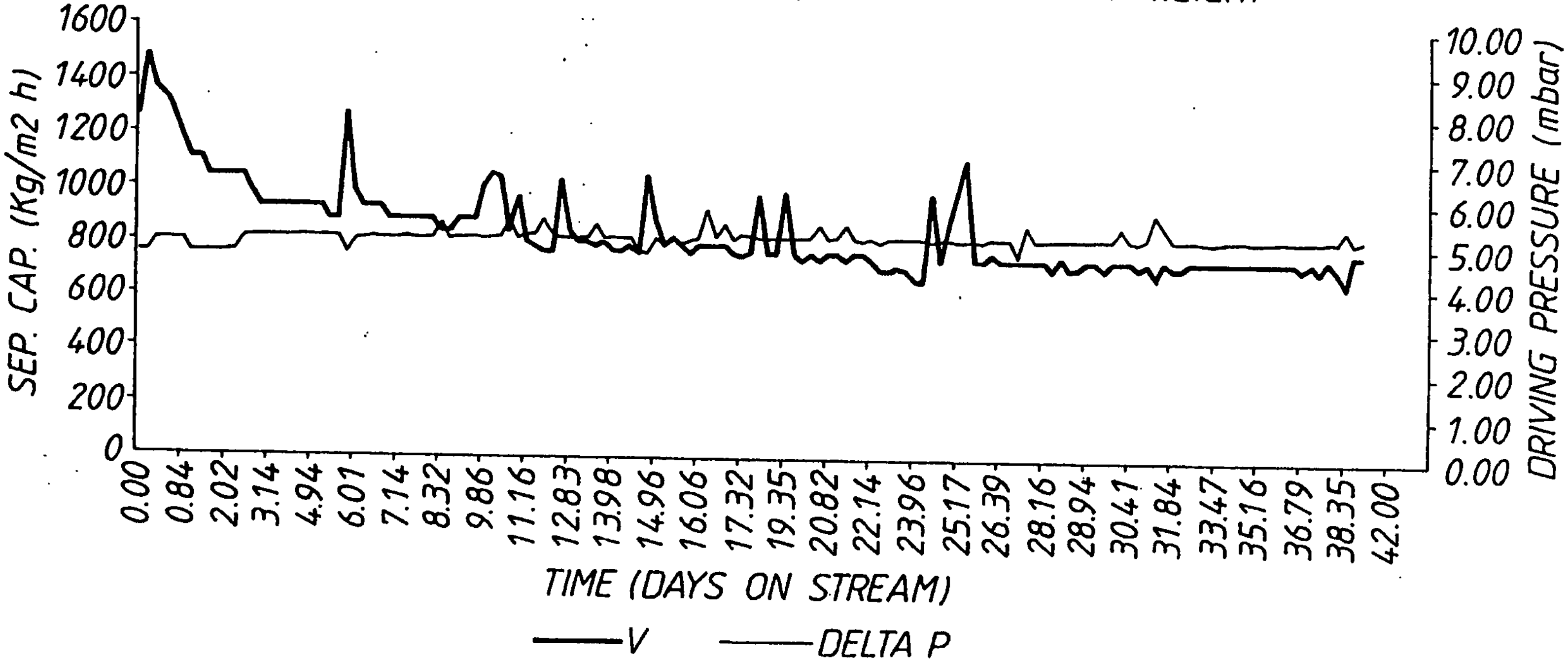

Fig.10

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: B01J 8/22

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: B01J, C07C, C10G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,P	WO, A1, 9316795 (DEN NORSKE STATS OLJESELSKAP A.S), 2 Sept 1993 (02.09.93), figure 1, claims 1,4,5,16, abstract --	1-25
X,P	WO, A1, 9316796 (DEN NORSKE STATS OLJESELSKAP A.S.), 2 Sept 1993 (02.09.93), page 7, line 6 - line 21, figure 1, claims 1-10, abstract --	1-25
A	EP, A2, 0450860 (EXXON RESEARCH AND ENGINEERING COMPANY), 9 October 1991 (09.10.91) --	1

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

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"A" document defining the general state of the art which is not considered to be of particular relevance

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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
11 May 1994	18 -05- 1994
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Johan von Döbeln Telephone No. +46 8 782 25 00

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP, A1, 0450859 (EXXON RESEARCH AND ENGINEERING COMPANY), 9 October 1991 (09.10.91) -- --------	1

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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WO-A1- 9316796	02/09/93	AU-A- 3649693	13/09/93
EP-A2- 0450860	09/10/91	AU-B- 632413	24/12/92
		AU-A- 7401591	10/10/91
EP-A1- 0450859	09/10/91	AU-B- 632412	24/12/92
		AU-A- 7401491	10/10/91
		US-A- 5157054	20/10/92

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